

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant	: Marco Mario Tivelli, et al.
App. No	: 10/554,075
Filed	: September 6, 2006
For	: SEAMLESS STEEL TUBE WHICH IS INTENDED TO BE USED AS A GUIDE PIPE AND PRODUCTION METHOD THEREOF
Examiner	: Mark L. Shevin
Art Unit	: 1793
Conf No.	: 2845

DECLARATION UNDER 37 C.F.R. § 1.132**Mail Stop Amendment**

Commissioner for Patents

P.O. Box 1450

Alexandria, VA 22313-1450

Dear Sir:

I, Alfonso Izquierdo Garcia, do hereby declare as follows:

1. I am a named inventor of the above-identified application. I am currently employed as Canada Quality Regional Director at TenarisAlgoma in Canada. I am an industrial engineer specialized in steelmaking processes and have a Master in Science specialized in foundry. I have been working with Tenaris for the last 15 years. I worked at TenarisTamsa, Veracruz, Mexico from 1995 to 1999 as a Product & Metallurgy Senior Engineer where I was responsible for the corrosion and high collapse laboratory. I then worked at TenarisDalmine, Dalmine, Italy from 1999 to 2001 as a Product & Metallurgy Senior Engineer. From 2001 to 2009, I then returned to TenarisTamsa as a Metallurgy Research & Development Manager. I have been Canada Quality Regional Director at TenarisAlgoma from 2009 to today. My activities have been related to the analysis of the customer specifications, materials testing and design, research and development of new alloys to meet customer requirements, intellectual

property activities related to patent requests as well as participating in the national and international congress related to materials and oil and gas trends and technologies.

2. I have reviewed the above-identified application, including the presently pending claims in preparation of this Declaration. References to the above-identified application are made with respect to the English translation of the specification filed on October 24, 2005.

3. I have reviewed the English translation of Japanese Patent 09-235617 ("Kondo"), which I understand has been used to reject the claims of the above-identified application. Below is a comparison of reheat quenching processing, such as those used in the examples in the present application, and of direct quenching processing as used in Kondo.

4. Kondo discloses a process of manufacturing seamless steel tubes by a method based on in-line heat treatment which allows energy saving (see, paragraphs [0005] and [0049] of Kondo). In particular, Kondo discloses that the seamless pipe after finish rolling can be directly water quenched, without cooling and subsequent reheating, to exploit the heat possessed by the pipe at the exit of the mill (see, paragraph [0002] of Kondo). After direct quenching, the pipe is subjected to off-line tempering (see, paragraph [0002] of Kondo). Figure 1 schematically illustrates Kondo's full heat treat process, and this process will be referred as direct quenching (DQ) hereafter.

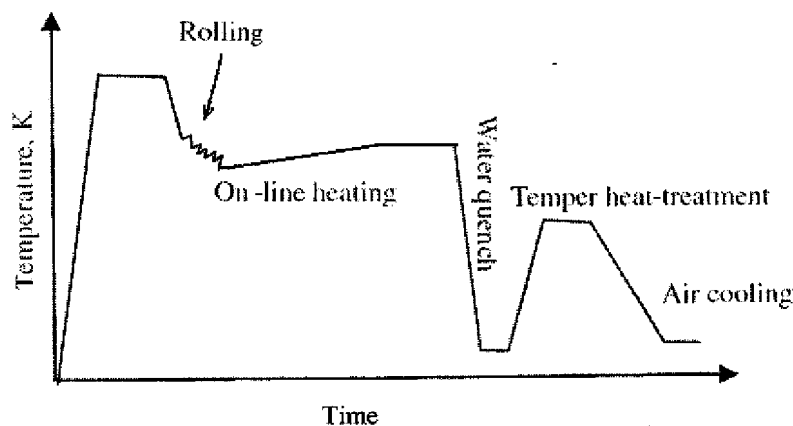


Figure 1

5. In contrast, the examples described in the above-identified application were quenched off-line. After hot rolling, the pipe is cooled in still air down to room temperature. Then, the pipe is subsequently re-heated to a temperatures higher than A_{c3} (about between 900 and 930 °C) and is then water quenched. The pipe is then tempered. Figure 2 schematically

illustrates the processing of the examples in the above-identified application, and this process will be referred as reheating quenching (RQ) hereafter. I believe that the above-described RQ process would be understood by one having ordinary skill in the art of steel pipe manufacturing based on the teachings of the specification, including but not limited to, page 24, line 4 to page 28, line 3.

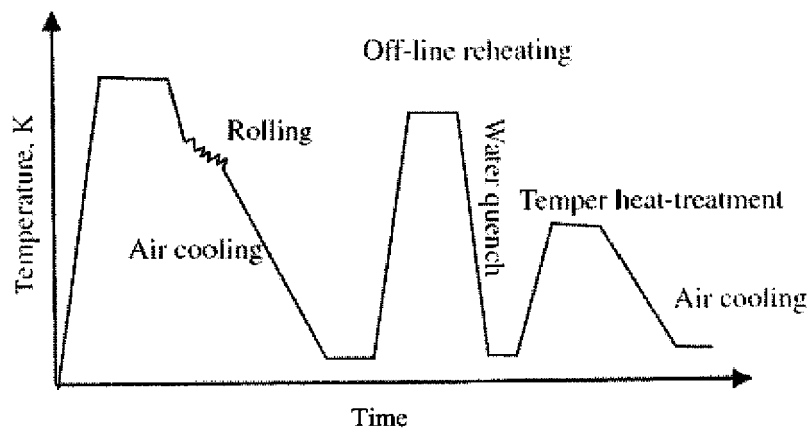


Figure 2

6. A seamless pipe manufactured by DQ such as described in Kondo has not been subject to transformation into ferrite and reverse transformation into austenite since it is not air cooled to room temperature after hot rolling and then reheated in the austenite field before quenching. Therefore, the austenite grains, which derive from the hot-working process at relatively high temperatures, are expected to be relatively coarse (e.g., large).

7. Kondo attempts to solve the problem of the relatively coarse austenite grain size produced by DQ by performing a hot-rolling reduction equal or greater than 40% in a temperature range between 1050 and 800 °C (see, paragraphs [0016], [0021], and [0053]-[0055] of Kondo). Hot rolling is performed in a furnace at the exit of the mill before quenching. After hot rolling, the tubes are not allowed to cool, but instead, the tubes are heated in a furnace at 850-1100 °C (see, paragraphs [0016], [0058], and [0059]). This further heat treatment promotes recrystallization of deformed austenite, and austenite grains are refined (e.g., grain size is reduced).

8. In contrast, RQ promotes the refinement of the austenite grains (e.g., reduces austenite grain size) through austenite-ferrite-austenite transformation. Refinement of austenite

grains through austenite-ferrite-austenite transformation can produce smaller austenite grains than the DQ of Kondo.

9. Samples 1-8 described below were produced using a RQ process. Sample 1 was manufactured with a pilot scale system (e.g. a system sized between a bench scale system and a full-size system). Steel with a composition listed in Table I was vacuum cast as an 80 kg ingot. The ingot was hot rolled by a pilot mill simulating a typical thermo-mechanical process of industrial seamless pipe mills for the production of linepipe with final wall thickness of 40 mm. The ingot was reheated at 1250 °C for 3 hours and hot rolled at about 1200 °C to 1040 °C using 8 passes for a total reduction of about 40%. The hot rolled material, after natural air cooling below 40 °C, was reheated in a muffle furnace at an austenitizing temperature of 920 °C (heating rate of 0.36 °C/s), held at 920 °C for 30 minutes, quenched in stirred water (cooling rate at mid-wall of 20 °C/s), and tempered at 660 °C for 40 minutes in another muffle furnace. The parameters of the process were controlled, and temperature of the steel was measured by a thermocouple imbedded at mid-thickness.

10. Samples 2-8 were produced at industrial scale by means of an electrical arc furnace to produce the steels. The steels were continuous cast into round bars, hot rolled to seamless pipes by retained mandrel mill process, and heat treated by walking beam furnaces equipped with gas fired burners. The continuous cast round bars were reheated to 1280-1300 °C, descaled, and pierced to hollows that were hot rolled similar to that of Sample 1. The pipes had a thickness from 38 mm to 42 mm. After the pipes were formed, they were cooled in still air down to 40 °C on a cooling bed and subsequently moved to the heat treatment plant. The pipes were then reheated by a walking beam furnace (heating rates of about 0.2 °C/s) to target temperatures of 900 °C to 930 °C, held for about 20 minutes, and rapidly moved to a quenching unit. Each pipe was at temperature above 820°C and rapidly rotated when it was dipped in a tank containing stirred water to make the heat transfer high and uniform and avoid pipe distortion. The water temperature was always below 40°C during quenching operation. After immersion for a time longer than 90 s, the pipes were taken and moved to the tempering furnace set at a temperature of 650 to 670 °C. The pipes were heated at a rate of about 0.12 °C/s, remained in the tempering furnace for about 200 minutes, and then cooled in still air on a cooling bed.

11. The compositions (weight percent) of each of the RQ Samples 1-8 are listed in Table I. The compositions were measured with a quantometer.

Table I:

Element	RQ samples (wt. %)							
	1	2	3	4	5	6	7	8
C	0.083	0.09	0.09	0.08	0.08	0.08	0.08	0.12
Si	0.23	0.29	0.29	0.30	0.28	0.27	0.27	0.29
Mn	1.28	1.28	1.28	1.19	1.16	1.13	1.13	1.21
P	0.007	0.012	0.012	0.008	0.010	0.011	0.011	0.014
S	0.0013	0.0011	0.0011	0.0007	0.0011	0.0020	0.0020	0.0020
Cr	0.10	0.16	0.16	0.10	0.24	0.12	0.12	0.18
Ni	0.38	0.30	0.30	0.45	0.40	0.37	0.37	0.38
Mo	0.15	0.15	0.15	0.14	0.14	0.13	0.13	0.14
Cu	0.12	0.11	0.11	0.12	0.10	0.15	0.15	0.19
Al	0.025	0.023	0.023	0.026	0.025	0.025	0.025	0.026
Nb	0.027	0.029	0.029	0.026	0.028	0.023	0.023	0.022
V	0.050	0.057	0.057	0.056	0.061	0.050	0.050	0.060
Ti	0.0012	0.013	0.013	0.016	0.013	0.002	0.002	0.003
Ca	0.001	0.0020	0.0020	0.0016	0.0021	0.0013	0.0013	0.0010

12. Austenite grain size of Samples 1-8 was measured at mid-wall by either the comparison procedure or the intercept procedure according with ASTM E112 standard on light microscopy images of transverse metallographic sections taken from the as-quenched material. The austenite grain boundaries were revealed by using an etching reagent based on saturated aqueous picric acid containing a wetting agent. The measurements were performed on 3 to 5 fields in the case of the comparison method, which involves comparison of the grain structure to a series of graded images, either in the form of a wall chart, clear plastic overlays, or an eyepiece reticle. Repeatability and reproducibility of comparison chart ratings are generally +/- 1 ASTM Grain Size number (G). The intercept method involves an actual count of the number of grains intercepted by a test line, per unit length of test line, used to calculate the mean intercept length. In the case of the intercept method, measurements were performed on 5 to 10 fields, having a total number of intercept from 400 to 500 on each specimen (for most grain structures, a total count of 400 to 500 intercepts or intersections over 5 to 10 fields produces better than 10 % relative accuracy). Relationship between the different grain size measurement methods that can be used according with ASTM E112, in particular between ASTM Grain Size No. (G) and mean intercept in μm are shown in Table II.

Table II.

Grain Size No. G	N_A Grains/Unit Area		\bar{A} Average Grain Area		\bar{d} Average Diameter		\bar{r} Mean Intercept		N_L No./mm
	No./in. ² at 100X	No./mm ² at 1X	mm ²	μm ²	mm	μm	mm	μm	
00	0.25	3.98	0.2581	258084	0.5080	508.0	0.4525	452.5	2.21
0	0.50	7.75	0.1290	129032	0.3592	359.2	0.3200	320.0	3.12
0.5	0.71	10.96	0.0912	91239	0.3021	302.1	0.2691	269.1	3.72
1.0	1.00	15.50	0.0645	64516	0.2540	254.0	0.2263	226.3	4.42
1.5	1.41	21.92	0.0456	45620	0.2136	213.6	0.1903	190.3	5.26
2.0	2.00	31.00	0.0323	32258	0.1796	179.6	0.1600	160.0	6.25
2.5	2.83	43.84	0.0228	22810	0.1510	151.0	0.1345	134.5	7.43
3.0	4.00	62.00	0.0161	16129	0.1270	127.0	0.1131	113.1	8.94
3.5	5.66	87.68	0.0114	11405	0.1068	106.8	0.0951	95.1	10.51
4.0	8.00	124.00	0.00806	8065	0.0898	89.8	0.0800	80.0	12.50
4.5	11.31	175.36	0.00570	5703	0.0755	75.5	0.0673	67.3	14.87
5.0	16.00	248.00	0.00403	4032	0.0635	63.5	0.0566	56.6	17.68
5.5	22.63	350.73	0.00285	2851	0.0534	53.4	0.0476	47.6	21.02
6.0	32.00	496.00	0.00202	2016	0.0449	44.9	0.0400	40.0	25.00
6.5	45.25	701.45	0.00143	1426	0.0378	37.8	0.0336	33.6	29.73
7.0	64.00	982.00	0.00101	1008	0.0318	31.8	0.0283	28.3	35.36
7.5	90.51	1402.9	0.00071	713	0.0267	26.7	0.0238	23.8	42.04
8.0	128.00	1984.0	0.00050	504	0.0225	22.5	0.0200	20.0	50.00
8.5	181.02	2805.8	0.00036	366	0.0189	18.9	0.0168	16.8	59.48
9.0	256.00	3968.0	0.00025	252	0.0159	15.9	0.0141	14.1	70.71
9.5	362.04	5611.6	0.00018	178	0.0133	13.3	0.0119	11.9	84.09
10.0	512.00	7936.0	0.00013	126	0.0112	11.2	0.0100	10.0	100.0
10.5	724.08	11223.2	0.000089	89.1	0.0094	9.4	0.0084	8.4	118.9
11.0	1024.00	15872.0	0.000063	63.0	0.0079	7.9	0.0071	7.1	141.4
11.5	1448.15	22448.4	0.000045	44.6	0.0067	6.7	0.0060	5.9	188.2
12.0	2048.00	31744.1	0.000032	31.5	0.0056	5.6	0.0050	5.0	200.0
12.5	2896.31	44992.9	0.000022	22.3	0.0047	4.7	0.0042	4.2	237.8
13.0	4096.00	63488.1	0.000016	15.8	0.0040	4.0	0.0035	3.5	282.8
13.5	5792.62	89785.8	0.000011	11.1	0.0033	3.3	0.0030	3.0	336.4
14.0	8192.00	126976.3	0.000008	7.9	0.0028	2.8	0.0025	2.5	400.0

13. Table III lists the minimum, maximum, and average values of the austenite grain size measured on various fields for Samples 1-8.

Table III:

	AGS (μm) of RQ samples							
	1	2	3	4	5	6	7	8
Maximum	14.1	15.8	16.2	17.5	17.9	15.9	16.1	16.4
Minimum	11.9	12.8	12.3	13.9	12.2	12.6	12.7	11.3
Average	13.0	14.0	14.9	15.9	14.1	14.8	15.1	13.4

14. Samples 9-12 were produced using a DQ process. Samples 9 and 10 were produced by a quenching and deformation dilatometer. This laboratory equipment is able to heat by an induction coil (heating rates from 0.05 to 20 °C/s) and cool by helium gas flow (cooling rate from 120 °C/s to 0.005 °C/s) under very controlled and reproducible conditions a steel cylindrical specimen. The dilatometer can also deform the specimen at high temperature by compression load and measure the length changes during the thermo-mechanical cycles. Samples 9 and 10 were taken from an industrial hollow (e.g. the round cast bar after piercing)

and heated at 10 °C/s to 1200°C and 1250 °C, respectively, maintained for 600 s, cooled at 3 °C/s at 1000 °C and 1050 °C, respectively, and deformed with reduction of 40%, direct heated for 600 s in order to maintain the sample at temperatures above Ac3 and quenched (cooling rate greater than 40 °C/s). I believe that this is representative of the DQ process of Kondo as a reduction of about 40% was given at temperatures close to or below 1050 °C and direct quenching was carried out.

15. Samples 11 and 12 were produced at industrial scale with similar process used for samples 2-8, but direct quenching was simulated. After hot rolling and heating in the intermediate furnace, a 300 mm long section was cut from the pipe and quenched in a water tank within 120 seconds of being removed from the intermediate furnace. Therefore, they were not subjected to air cooling down to 40 °C and subsequent reheating and quenching. Samples 11 and 12, which were taken from the same pipe along a given circumference at a relative angular distance of 45°, I believe are representative of the DQ process of Kondo as an average reduction of about 40% was given at temperatures close to 1050 °C and direct quenching was reproduced. The pipes had a wall thickness of about 24 mm. The compositions of each of the DQ samples are listed in Table IV.

Table IV:

Element	DQ samples (wt. %)			
	9	10	11	12
C	0.08		0.10	
Si	0.27		0.26	
Mn	1.30		1.08	
P	0.013		0.014	
S	0.0025		0.0025	
Cr	0.10		0.14	
Ni	0.30		0.32	
Mo	0.12		0.13	
Cu	0.15		0.12	
Al	0.030		0.025	
Nb	0.029		0.026	
V	0.060		0.058	
Ti	0.020		0.003	
Ca	0.0012		0.0014	

16. Austenite grain size of these DQ Samples 9-12 was measured by the same technique as used for RQ Samples 1-8 described above. Table V lists the minimum, maximum, and average values of the austenite grain size measured on various fields for Samples 9-12.

Table V:

	AGS (μm)			
	DQ samples			
	9	10	11	12
Maximum	36.3	60.8	39.2	59.2
Minimum	27.2	52.2	32.1	51.1
Average	30.3	56.1	35.0	54.0

17. The average grain size of the RQ Samples 1-8 falls within the austenite grain size ranges described in the specification as 12 to 20 microns (see page 14, lines 7-10 of the specification) and/or from ASTM Grain Size No. 9 to 10 (e.g., from 14.1 μm to 10 μm according to Table II (see circled columns)) (see page 21, lines 17-18 of the specification). I believe that a person having ordinary skill in the art would understand that the grain size ranges described in the specification mean "average grain size" measured according with ASTM E112 standard.

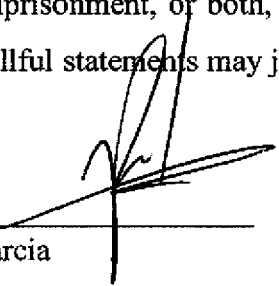
18. As listed in Tables III and V, the average austenite grain size obtained from DQ was 30.3 μm to 56.1 μm and from RQ was 13 μm to 15.9 μm . Therefore, based on the differences between the process of DQ and RQ discussed above as well as the experimental data, I believe that tubes produced by the DQ of Kondo would not be expected to have an average grain size ≤ 20 microns.

19. Moreover, the method disclosed in Kondo would be difficult to use to manufacture heavy wall steel pipe (wall thickness ≥ 30 mm). For example, as reported in EP1876254A1 Kondo et al. (see paragraph [0007]) for pipes with thickness greater than 30 mm it is difficult to perform a hot-rolling reduction equal or greater than 40% in a temperature range between 1050 and 800 °C.

20. The above-identified application is the national phase application of International Publication No. WO 2004/097059 ("WO '059") which published in Spanish. I am fluent in Spanish and English. At page 25, lines 4-10 of the specification of the above-identified application (corresponds to page 25, lines 11-18 of WO '059), each instance of the word "tempering" should read "quenching."

Application No.: 10/554,075
Filing Date: September 6, 2006

21. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful statements may jeopardize the validity of the application or any patent issued thereon.

By: 
Alfonso Izquierdo Garcia

Date: March 23rd, 2011

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